

CEMENT AND LIME MANUFACTURE

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Closed-Circuit Grinding as an Engineering Problem.

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FOR some twenty to twenty-five years the cement industry experimented with various designs of mechanical air separators in view of the thorough development of closed-circuit grinding in the metallurgical industries, but the results were not sufficiently attractive to make their adoption universal. As a result of considerable research and application, however, air separators (Fig. 1), properly designed and correctly applied, offer great opportunities for improvement in cement quality and in reduced grinding costs. At the present time, one company alone has installed more than 170 air separators in its cement plants, and these installations also make possible the manufacture of Portland cements of any surface area including high-early-strength cements with less power and a substantial decrease in grinding temperatures. Closed-circuit grinding enables cement to be produced with surface areas up to 3,323 square centimetres per gramme; this has not been achieved by any other method. The demand for air separators in clinker grinding is increasing due to the higher requirements for the surface area of cements; for example, one cement company now has more than fifty in its various plants. By the use of air separators, higher tensile and compressive strengths can be obtained at little extra cost, while in producing normal Portland cements, with surface areas of about 2,000 square centimetres per gramme, the capacities have exceeded those of open circuit grinding.

Principles of Grinding in the Cement Industry.

Even to-day the cement grinding process is not standardised. Tube mills predominate, either of the unit or compartment type, the latter seldom requiring a preliminary grinder. In other cases, particularly on raw material, several types of preliminary grinders are used for the first reduction, followed by a tube mill or other finishers. With the advent of closed-circuit grinding methods

for both raw material and clinker came the use of screen and air separators to give increased capacities, and more uniform and finer outputs and to obtain high early strength by giving attention to changes in the raw material mix; also the study of the effect of raw material fineness to the kilns, the degree of burning, and the ability to carry a high lime ratio. All these are steps in the direction of grinding the clinker finer, and in many cases enable high-early-strength cements to be produced by increasing the percentage of fines between a given range rather than by increasing the fineness of the impalpable powder beyond a limit that has little, if any, effect on the final product.

The application of the method involves a knowledge of the cement manufacturing process from both a chemical and a physical standpoint and an operating



Fig. 1.

experience with tube mills and preliminary machinery, as a perfect balance must be reached and maintained between all equipment within the closed-circuit cycle. The amount of feed, the circulating load, the percentage of fines in the product (and in the separator tailings returned) of each contributing machine, the ball load and the sizes of grinding media and diaphragms in the tube mills, and the screens in the pulverisers, all have to be predetermined accurately to gauge the results to be expected. For super-strength cement even the heat of grinding must be lowered.

Open-Circuit Grinding.

Open-circuit grinding, sometimes known as "one-passage" grinding, requires a pulveriser to finish all the material fed to it in one passage through. The mills are required not only to grind but also to size the material, discharging the

product only when it is reduced to the finished state. In this method the pulveriser must grind to a finish the entire amount fed to it. The amount fed is exactly equal to the amount discharged; if it were fed too fast the product would be too coarse and the mill would become "plugged"; if it were underfed, the output would be finer than necessary. In such circumstances, when estimating the feed rate, the physical characteristics of the material being ground, its size, hardness, toughness, moisture content, etc., must be considered, for if any of these vary so will the capacity of the mill and the fineness of the product.

If a tube mill is fed with material 90 per cent. of which passes a 100-mesh screen and 10 per cent. passes a 4-mesh screen, and if a 100-mesh product is desired, the feed must be adjusted so that the 10 per cent. of 4-mesh particles will be ground to 100-mesh size before any material is allowed to discharge. That proportion which is already fine enough cannot readily escape until 10 per cent. of 4-mesh material is ground to a like fineness. It is obvious that more than 90 per cent. of the grinding ability of the mill is penalised, for the 90 per cent. of fines prevents the action of the pebbles or balls which are cushioned, coated, and obstructed by these fines. This is reflected in curtailed output, excessive wear and power consumption, higher initial costs and extra space occupied by several mills whose work could be done by one. If the feed and discharge rates are not exactly equal, any variation is immediately reflected in the fineness of the product. If the physical characteristics of the material vary, the rate of feed must be changed or the quality of the output will vary. The only method of correcting the variables is to make constant screen analyses of the discharged product and modify the feed rate. Quick corrections are not practical and usually result in the operator underfeeding the mill to be sure of the fineness, with the result that output is reduced and the material is ground finer than necessary.

Closed-Circuit Grinding (with a Separator).

Closed-circuit grinding is a method of reduction in which the pulveriser is operated in closed circuit in connection with a classifier (wet, dry, air or screen), in which case the mill is not required to finish all the material fed to it to the desired fineness in one passage through. The sizing is done by the classifier; the only duty of the mill is that of pulverising. The total discharge from the mill (coarse and fine material) passes through the classifier, which selects that portion that is of the required fineness and rejects and returns the remainder to the mill for further reduction with the fresh feed. The portion returned to the mill is termed the "circulating load."

In the open-circuit method the feed rate was limited to the ability of the mill to grind the hardest, largest, or dampest particles. In the closed-circuit method the full ability of the mill is used to grind only; it need not finish in one operation the whole of the feed. The circulating load may be—and it is desirable that it should be—several times that of open-circuit feed, or such that a screen analysis of the product shows as little as 50 per cent. of the desired

fineness. This means that if by open-circuit methods the rates of feed and the discharge are five tons per hour of finished product, by closed-circuit methods the rate of feed, including the circulating load, may be increased many times. For instance, if it were doubled and the mill ground only 80 per cent. of the material passing through, the capacity would be eight tons of finished product. Again, if the feed rate plus the circulating load were increased to fifteen tons per hour and the mill ground only 70 per cent., its output would be $10\frac{1}{2}$ tons per hour. This would require no more power beyond that required by the classifier, which is a small percentage of the mill power. Cases have occurred where a ten-fold increase in circulating load has shown economical results.

Fig. 2 is a grinding diagram of a standard 6-ft. by 26-ft. tube mill operating under open-circuit methods, and shows the relative pulverising effect upon the material at each foot of length traversed. It is seen that 60 per cent. of the

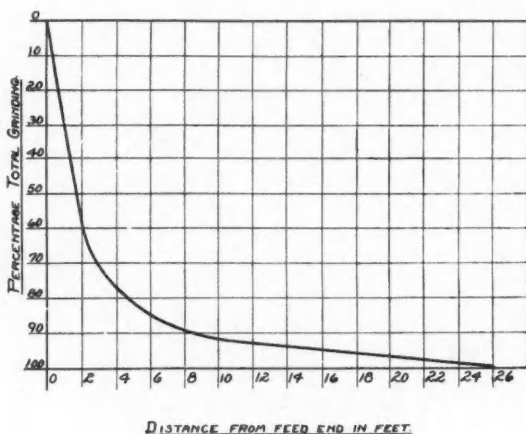


Fig. 2.

grinding is accomplished in the first 2 ft., 75 per cent. in the first 4 ft., 85 per cent. in the first 6 ft., and 90 per cent. in the first 8 ft., and the remainder of the tube mill (18 ft.) is used to finish 10 per cent. of the material fed. In other words, 30 per cent. of the length grinds 90 per cent. of the material, while 70 per cent. grinds only 10 per cent. It is not difficult to forecast the result if 18 ft. of the tube mill were removed and the first 8 ft. only were used in closed circuit with a classifier. If the 26-ft. mill ground ten tons of material per hour, then the 8-ft. section would grind nine tons, 70 per cent. of the mill power would be saved, and a similar percentage of upkeep, floor space, etc., would be saved. If the feed were increased and the circulating load of the 8-ft. section built up until it finished only 70 per cent. of the feed instead of 90 per cent. as formerly, its output would be increased to 70 per cent. of the feed, including the circulating

load. Thus if these loads were doubled (20 tons per hour), the mill capacity would be increased to 14 tons per hour. The only additional power required would be that taken by an elevator and air separator (approximately 25 h.p.). The tube mill need not be reduced in length to work efficiently, for by using the full length and increasing the feed, and by using the mill to grind only and not to finish (depending on the separator to regulate fineness), the character of the graph will be changed and the efficiency will be distributed much more evenly throughout the entire length of the mill.

Any mill will reduce a portion of its output to any fineness, and the separator classifies the product and removes the fines of any reasonable size. The balance is reground, and it does not matter how many times this unground or partially ground material passes through the mill before it is reduced to the required fineness, because it will eventually be ground and removed by the classifier without affecting the pulveriser.

The principle of closed-circuit grinding is the use of two independent machines working in close co-operation with each other. The function of the first is to grind to maximum capacity, unhindered by cushioning fines, by overgrinding, by withholding the fines until the coarser particles are reduced, by restricting output to a definite mesh, by plugging, or by wear of internal screens. The separator is large enough to handle any reasonable circulating load, its output is unaffected by coarse particles, excessive load, unusual dampness, or the amount of fines in the material fed to it. It may be quickly and accurately adjusted for modifying products within a wide range, it requires no attention, it is durable, and it requires a small amount of power. The pulveriser and classifier function as a balanced unit.

TABLE I

THEORETICAL TABULATION OF CLOSED VERSUS OPEN-CIRCUIT GRINDING ASSUMING THAT SELECTOR EFFICIENCY IS 100 PER CENT.

Tube mill. Size (ft.)	Horse power, mill only. Open circuit	Horse power, mill only. Closed circuit	Additional Power. Selector unit	Total power	Fines in mill discharge(%)	Circulating load. Tons per hour	Finished output. Tons per hour	Power per ton of output
7 by 24	500			500	100	13	13	38.4
"		500*	45	545	90	26	23.4	23.3
"		"	"	"	80	39	31.2	17.4
"		"	"	"	70	52	36.4	15.0
"		"	"	"	60	65	39.0	13.9
"		"	"	"	50	78	39.0	13.9

* The power, in practice, is less as the circulating load increases, because due to the speed at which the material passes through the mill there is less load in the mill at any time.

Increased capacity is accomplished without extra power except that due to the separator, and power costs per ton of output are decreased by closed-circuit grinding; Table I illustrates this point. A 7-ft. by 24-ft. tube mill in a cement plant grinding limestone is used as an example. This mill, formerly operated in open circuit, had a capacity of 13 tons per hour grinding to a fineness of 97 per

cent. passing a 100-mesh sieve with an expenditure of 500 h.p. The table shows the output and power used by the same tube mill grinding to the same fineness in closed circuit with a selector. It will be noted that by increasing the circulating load from 13 to 26 tons the mill discharges a product of which only 90 per cent. is of the required fineness, but twice as much material has passed through the system in the same time, with the result that the total output of finished product is 23.4 tons instead of 13 tons by the open-circuit method; although the power used by the selector unit (45 h.p.) has been added, the power required per ton of output has dropped from 38.4 to 23.3 h.p. By further increasing the circulating load a progressive increase in output and decrease in power are obtained until a maximum economy is reached—when the load to be pulverised reaches 65 tons per hour, or five times the open-circuit feed rate, when the capacity is 39 tons per hour with a total power expenditure of 13.9 h.p. per ton. The next increase of circulating load shows no saving in power and no increase in output, so that there is no advantage in further increasing the circulating rate.

Although these figures are theoretical and based upon a selector efficiency of 100 per cent., in practice a minimum increase in output of 50 per cent. and a maximum of 200 per cent. may be obtained, depending upon the equipment used, the material being ground, the fineness desired, and the arrangement of the plant. The advantages are: (1) Large increase in output; (2) Uniformity of product; (3) Fineness of product; (4) Complete control of output; (5) Saving of labour. Another important advantage of closed-circuit grinding is the cooling effect of large circulating loads passing through a grinder and selector. Heat generated by the crushing and friction of the grinding media is quickly dissipated by the great volume and steady flow of the material passing rapidly through the system. The cooler the mill the less the cost of upkeep and power required. In the reduction of cement clinker by closed-circuit methods the temperature is reduced by 50 to 100 deg. F. The moisture content is also reduced, and much damper materials may be ground in closed circuit than in open circuit, especially if an air separator is used. A mill operates with less power under closed-circuit methods than by open-circuit processes, because the material does not accumulate within the grinding chamber; the material is passed through at high speed and discharged freely, so that there is less in the mill at any time. This is particularly true of tube mills and of the roller or hammer types which use internal screens or close grate spacings. With the latter types coarser screens or wider grate spacings are used to assist the discharge, and no attempt is made to require the mills to finish all the material within themselves.

Applications of Closed-Circuit Grinding.

Most finishing mills require a preliminary grinder to provide material of a size upon which they can work most effectively. The preliminary grinder may be operated in closed circuit with a classifier, independently, or in connection with the finishing mill. Each may use the same classifier, depending upon the result desired. For instance, if a hammer mill is used as a preliminary grinder

to a tube mill, and the final output of the tube mill is to pass a 100-mesh sieve, then one of four methods can be used :

(1) Hammer mills produce a large percentage of material passing a 100-mesh sieve at the first "break." They can grind to 80 or 90 per cent. passing a 20-mesh sieve economically, but they cannot successfully finish their own tailings finer. The fines produced may be effectively operated in closed circuit with an air separator which will remove the 100-mesh material and thus relieve the tube mill of the effort of over-grinding the material which is already sufficiently reduced.

(2) The same separator may be used in closed circuit for both hammer and tube mill.

(3) An independent separator may be used for each mill.

(4) A screen and separator may be used after the hammer mill. The screen is installed to supply the separator with a product of 16-mesh and finer, from

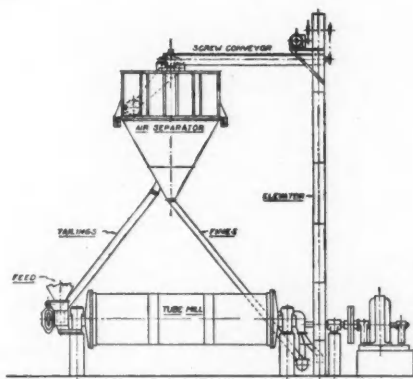


Fig. 3.—Closed-Circuit Unit with Tube Mill and Air Separator.

which it removes the material passing a 100-mesh screen and finer and returns the material between 16 and 100-mesh screens to the tube mill ; the rejects from the screen (plus the 16-mesh material) are returned to the hammer mill for further reduction.

The same air separator can also be used for the tube mill (*Fig. 3*). In such an installation the hammer mill grinds the whole of the material to 16-mesh fineness in closed circuit with a screen, and at the same time the 100-mesh material is reclaimed from its product. The rejects from the air separator (between 16 and 100 mesh) are an ideal feed for the tube mill, and the latter, working in closed circuit with the same air separator, is working most effectively. Similar installations can be made with other preliminary grinders, depending upon whether they make sufficient finished fines to remove at that stage, or whether a combination of air separator and screen is desirable.

Lime in the Leather Industry.

FROM THE BRITISH LEATHER MANUFACTURERS' RESEARCH ASSOCIATION.

THE treatment of hides and skins with a suspension of lime is one of the essential processes in the manufacture of leather prior to tanning. The first object of liming is to unhair the hides or skins. The second is to prepare the pelts for tanning. It is therefore of importance that the type of lime most suitable for this particular purpose should be used.

Lime contains a certain number of impurities, mainly in the form of iron and aluminium ($\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$), silica (SiO_2), magnesia (MgO), and sulphates. A pure lime, that is one consisting mainly of calcium oxide, should be used in the tannery. A low content of iron and aluminium, magnesia, and sulphates is desirable, i.e. the lime should not contain more than 0.4 per cent. of any of these compounds. The silica content is not of great importance, at any rate up to a concentration of 2 per cent.

Either quicklime or hydrated lime can be used by the tanner in making up his lime liquors. At present quicklime is more generally used.

Care must be taken in slaking quicklime for use in the lime yard to avoid a dry crumbling and local "burning" of the lime. This is liable to occur if the lime is slaked by allowing a slow trickle of cold water to fall on the lime. Granular lumps of useless lime are formed, the larger of which fall to the bottom of the slaking vessel and are thrown out with the stones and other gross impurities. There is also a danger of the smaller granules of such improperly slaked lime causing lime burns by continuing to slake slowly when in contact with hide or skin. Lime for use in the tan yard should be slaked in a vessel containing sufficient hot water to submerge the whole of the lime. The lime should then be added cautiously so as to allow gentle boiling. Small amounts of cold water may be carefully added if violent boiling with splashing and loss of lime occurs. If the goods are to be unhaired by pasting the flesh side with a lime "paint," the thick paste of lime is mixed with small quantities of sulphide of sodium or arsenic. If the goods are to be unhaired by complete immersion in a lime suspension, then to the thick lime paste formed by slaking is added the necessary volume of cold water.

The use of hydrated lime has made progress recently in the industry, but certain steps have to be taken to ensure results equal to those of quicklime. The most important of these is to keep the suspension of hydrated lime in water for two days before use. As regards standards of purity, the same apply for hydrated lime as for quicklime.

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The Design and Operation of Modern Lime Works—II.*

By N. V. S. KNIBBS, D.Sc.

The Size of Limestone for Lime-burning.

THE size of stone used in lime burning influences the design of the whole of the burning plant. There is considerable variation in practice, depending upon the nature of the stone, the methods used in quarrying, the size of the quarry operations, and the facilities for the disposal of small stone for other purposes. As mentioned in the previous article there are few data available on the rate of burning of stone, and the effect of stone size on that rate, and those few data are not in agreement. Nevertheless they are at least some guide and are therefore given graphically in *Figs. 2 and 3*.

The time in hours required for complete calcination of limestone is plotted against the temperature of the surroundings (the burning temperature), and separate curves are given for 10, 6, 4, 2 and 1-inch lumps. They apply strictly only to spheres of the diameter shown, but an irregular piece of stone will calcine at about the rate of a sphere of diameter equal to the least of the axes of the lump. It should be noted that limestone actually burnt seldom has an effective size of more than 6 in., because the smallest axis of a piece that a man can lift is seldom much more than 6 in. and also because fissures in the stone have the effect of reducing the distance the heat has to travel through lime and stone.

The figures plotted in the curves have been calculated by the different observers from experimental results obtained by them, and in all instances the curves for larger sizes depend on extrapolation. It will be seen that Haslam and Smith's calcination times are very much shorter than those of other observers, and they cannot be applicable to actual burning conditions. For example, they show that a 6-in. lump will take only about three hours to calcine at 1000 deg. C., which is obviously incorrect. The results of Furnas, too, are much too low at high temperatures, as, for example, when he finds that a 10-in. lump will take only about two hours to calcine at 1300 deg. C. Block's figures are not equivalent to the others, because they apply to kiln conditions where the stone is preheated at a lower temperature. They are pure calculations, and err in being too long due to their being based on incorrect data. In spite of the variation in the figures it is obvious that, purely from the standpoint of calcination, the smallest stone is the best. Why, then, is nearly all lime made in England in the form of large lumps? There are four main reasons which will be considered briefly.

In the first place large stone generally costs less to produce than smaller stone, and this is especially so in the smaller plants where stone is quarried for lime-burning only. In larger quarries it would seldom be a predominant factor, and where there is a primary crusher taking large pieces it may be as economical to produce smaller stone.

* Continued from January, 1937.

Secondly, the kilns in use to-day, with the exception of the rotary kiln and a few isolated instances of kilns built for special purposes, are designed for large stone and will not operate on small stone. To burn small stone therefore requires a new departure in kiln design which has not yet been worked out except in the case of the rotary kiln.

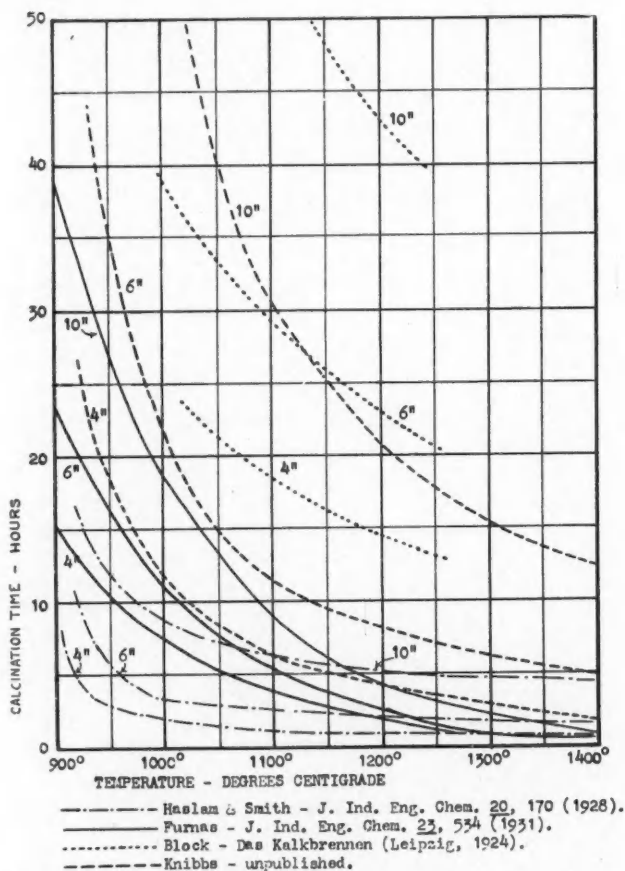


Fig. 2.—Rate of Calcination of 4 in., 6 in. and 10 in. Limestone.

Thirdly, nearly all lime requires sorting to separate underburnt, overburnt, and impure lime, or to pick out good lime from the run-of-kiln material. Sorting is very much easier when the lumps are large and, as size is reduced, a point is soon reached when it is uneconomic. This is perhaps the most cogent reason for burning in large pieces.

Lastly, the users of lime demand it in large pieces. With very few exceptions there is no valid reason for this, and actually it would be easier to use if supplied in small pieces. It has arisen merely because good lime has always been supplied in lumps, and in the past small lime has ordinarily been impure and overburnt. It would not take much persuasion to induce users to take small lime of uniform

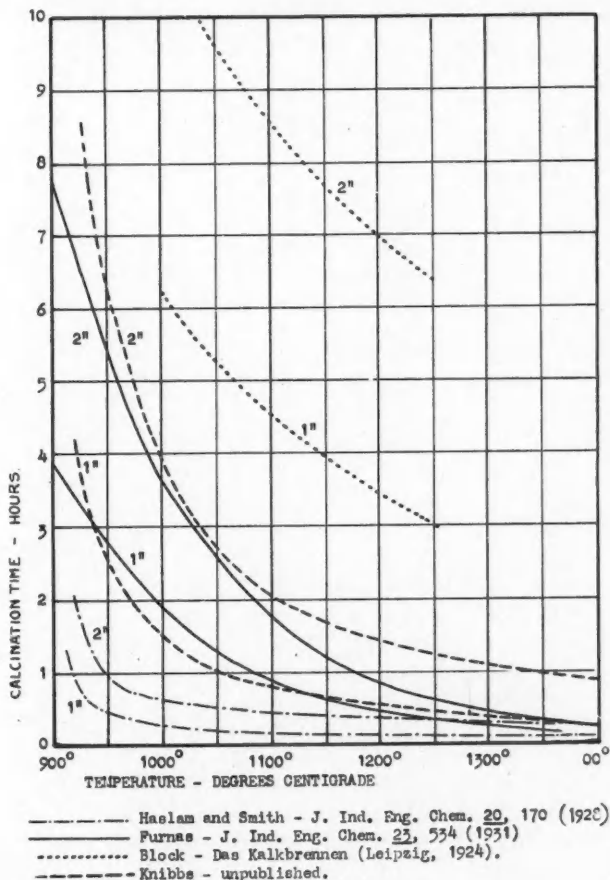


Fig. 3.—Rate of Calcination of 1 in. and 2 in. Limestone.

high quality, in the same way as pebble lime from rotary kilns has been adopted in the United States, where rotary kilns were at first employed as a means of using up "spalls." Their economic success, in spite of their low fuel efficiency, later resulted in the installation of plants in which all the stone was reduced to small size and rotary kilns alone used for burning. In England there is generally

a market for small stone and it is sold as such, but small soft stone and chalk are not much in demand, and there is already a need for burning plant to produce lime from such small stone. Rotary kilns have been considered here to deal with the problem, but the large output of a rotary kiln of what is considered economic size, and the element of novelty in their use in this country, have hitherto, with one exception, confined their use to the calcination of calcium carbonate sludge and there is a real need for a kiln of moderate size that will burn small stone economically. The problem will be fully considered later.

Sizing Stone for Burning.

Limestone for burning generally varies widely in size and it is very seldom that it is closely graded. The upper limit of size is often the heaviest piece that a man can load unaided, and the lower limit is frequently fixed by draught considerations in the kilns, the use of too much small stone decreasing the draught unduly. In blastfurnace practice the use of a high-pressure blast permits the use of a greater amount of small stone than in a lime kiln, but recent investigations have shown great benefits from grading the burden and in a lime kiln those advantages must be still more pronounced. Sizing of stone for lime burning is therefore worthy of much more attention than it receives at present.

When a kiln is filled with evenly-sized stone the percentage of voids is at a maximum and the resistance through the kiln at a minimum for stone of that average size. If natural draught is used with evenly-sized stone the kiln will burn more lime, and if induced or forced draught is employed the power consumption will be at a minimum. The draught will be uniform over the whole area (except against the walls, where the voids are greatest) whereas with a charge of stone of mixed size there may be a tendency to "channelling." If the kiln is gas-fired the gas will penetrate farther through the lime and spread more evenly across the kiln, with consequent greater equality of heating. There is also less chance of recarbonation, a trouble often experienced in mixed-feed kilns of low output and in some gas-fired kilns. Recarbonation will be discussed more fully later.

The most important advantage of stone of uniform size is in the quality of the lime produced. Even when a kiln is heated uniformly over its whole cross-sectional area it will not produce uniformly-burnt lime unless all pieces are of the same size. If the size varies the small pieces will be overburnt before the large pieces are completely calcined, unless the temperature is very low in which case the time required by the large pieces becomes excessively long. Close grading of the stone is therefore an important aid to high quality.

A practical objection to the sizing of stone for lime burning is that it costs more to produce stone of uniform size, and another use must be found for the other sizes that are necessarily made at the same time. If the output is large enough the ideal arrangement is to separate the stone into different sizes and have a kiln specially designed for each size, a method of manufacture that would give lime of a quality and uniformity superior to any results achieved under present practice and with lower fuel consumption. Unfortunately there are

very few plants in which the output would justify so much separation, but a compromise is possible that will achieve at least some of the benefits of separate burning.

If a mixed-feed kiln is charged with graded stone in layers varying in thickness according to the amount of each size, and each with its appropriate admixture of fuel, it will operate like a series of separate kilns having the open spacing (high percentage of voids) of the kiln charged with stone of uniform size. It will not be as efficient as a number of separate kilns, because the rate of burning must be the same for all sizes, whereas each size should operate at its optimum rate (which optimum varies with size), but it will be a distinct improvement over the ordinary method of using unsized stone. The same procedure is not well adapted to gas firing, because the means for causing uniform spreading of the gas must vary with the size of the stone. A good combination would be to burn the larger sizes of stone in a gas-fired kiln and the small stone, after it has been graded into sizes, in a mixed-feed kiln.

Recarbonation.

Recarbonation, or the reabsorption of carbon dioxide by quicklime, has a profound effect on the properties of lime, and as there is some misunderstanding about the phenomenon a short discussion here may be of value. It has often been stated that recarbonation of lime is due to the absorption of carbon dioxide from ordinary cold air, and "air slaking" and "recarbonation" are sometimes regarded almost as synonyms. This is a fallacy. At ordinary temperatures lime (calcium oxide) will not combine with carbon dioxide, even slowly. It must be changed to the hydroxide by absorption of water before it will react, and therefore it is only the air-slaked powder that combines with carbon dioxide. In this climate ordinary air in summer contains about twenty times as much water as carbon dioxide by weight, and consequently when a piece of lime has been subjected to enough air to hydrate it completely it will have absorbed only about 1.6 per cent. of carbon dioxide as a maximum. Ultimately, on long exposure, a large part of the hydrated lime powder will have changed to carbonate, but this material, which is in powder form, is not the material generally known as recarbonated lime.

The recarbonation of lime is due to its exposure at elevated temperature, and generally above 500 deg. C., to gases containing carbon dioxide. Above 500 deg. C. lime absorbs carbon dioxide rapidly, the rate of absorption increasing up to the dissociation temperature. Recarbonation will therefore take place in the cooling zone of a kiln when there is any downdraught, and in open-top kilns a downdraught may easily occur. Recarbonation may also take place in a mixed-feed kiln in which the material is following down unevenly, so that the gases from the combustion of the fuel in one part diffuse over to another part where the fuel is all burnt and the lime cooling. In a gas-fired kiln it may take place to some extent where the gases impinge on the lime, especially if the gases are cool and high in carbon dioxide. It is, however, most frequently to be observed in short open-top kilns.

The main effect of recarbonation, even when it amounts to only 2 or 3 per cent.

carbon dioxide, is to reduce the reactivity of the lime and the rate of slaking. It therefore resembles overburning in its effects, and recarbonated lime, in fact, is often mistaken for overburnt lime.

The Calcination of Small Stone.

To show more clearly the effect of size of stone on the time required for complete calcination, *Fig. 4* has been prepared from some of the available data.

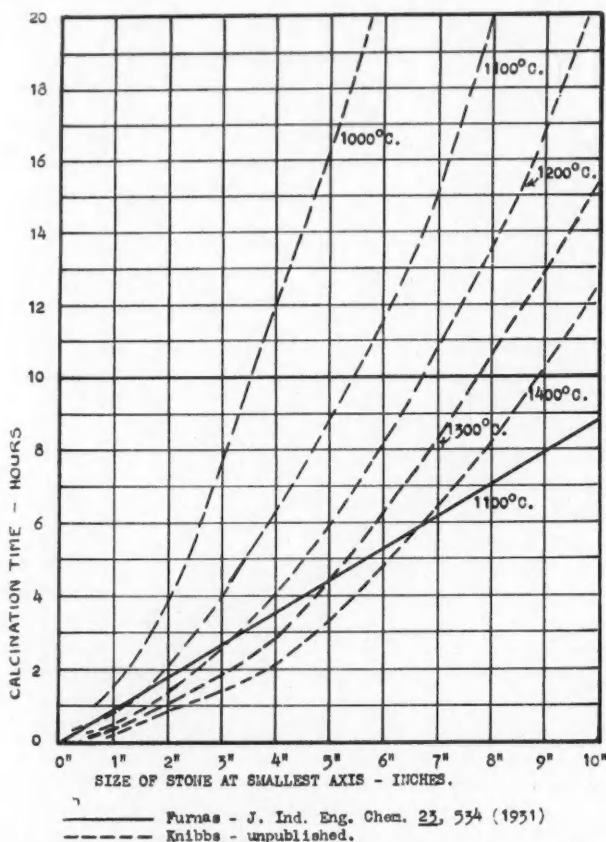


Fig. 4.—Rate of Calcination of Limestone—Variation with Size.

Caution is necessary in applying any of these laboratory data to practical lime-burning conditions, but if the curves are even very roughly correct the conclusions to be drawn from them are important and perhaps somewhat surprising. In a vertical kiln 6 in. may be taken as the ordinary size of stone, and 1150 deg. C. is a usual average temperature when burning fairly pure stone (involving a

maximum temperature of about 1400 deg. C.). The calcination time is therefore about nine and a half hours. If the same stone is crushed to 1 in. and calcined at an average temperature of 1050 deg. C. (maximum 1200 deg. C.) the time of calcination will be only about seventy minutes, or about one-eighth as long. Therefore the calcination zone of a kiln to burn 1-in. stone need be only about one-eighth as long as in a kiln to burn 6-in. pieces, provided the necessary heat can be generated in this short section. A lower burning temperature is assumed for the small stone, because it is easier to overburn it and because, in a mixed-feed kiln, it will naturally have a lower temperature on account of the more rapid rate of absorbing heat from the burning fuel.

Because of the much greater area exposed to the gases (the area is inversely proportional to the size of the stone), the good heat exchange conditions caused by the smaller and more numerous interstices through which the gases have to pass, and the short distance the heat has to travel within the lime and stone, there is a similar ratio in the preheating and lime-cooling zones of the kiln, and the conclusion is therefore reached that to burn 1-in. stone a vertical kiln of given cross-section need be only about one-eighth the height of a similar kiln to burn 6-in. stone, provided the requisite heat can be generated in the smaller space allowed. On the same basis 2-in. stone will require about three-tenths the height of 6-in. (or less if a higher burning temperature than the 1200 deg. C. maximum is assumed).

It might at first be thought that the higher resistance to the passage of gases offered by small stone would render a shaft kiln impracticable, but the resistance through unit depth of stone is approximately inversely proportional to the size of the stone and therefore the resistance will be rather less through the short kiln burning small stone if the ratios mentioned hold good. On account of the lesser height there will, however, be much less natural draught, and therefore, in practice, induced draught of some kind may be essential.

When the rate of calcination of still smaller stone is considered, the time required becomes so short that other methods of calcination become possible, where the total heating period is measured in minutes. Various methods of calcining small stone will now be considered.

The Sintering Machine.

Sintering machines were developed to convert fine iron ore and other ores into lump form by incipient fusion. The material is spread evenly on a travelling grate which passes under a refractory arch. Heating is by flame or by flame together with fuel spread on the top of the material to be sintered, and the hot gases are sucked downwards through the material. When burning small limestone by this method it was found necessary to use some solid fuel to supplement the flame, and also to spread a layer of burnt lime under the stone being burnt so as to avoid the necessity of having a burning temperature actually on the pallets of the grate. Small limestone has successfully been burnt on machines of this kind, but the fuel consumption is high and the capacity of the machine much less than when it is employed for sintering. This is inevitable, because of the

large heat absorption at high temperature involved in the lime-burning reaction as compared with sintering. At best, this type of burning plant demands stone in a finely divided condition and it is seldom that such stone is available commercially. Similar machines have been developed for the production of Portland

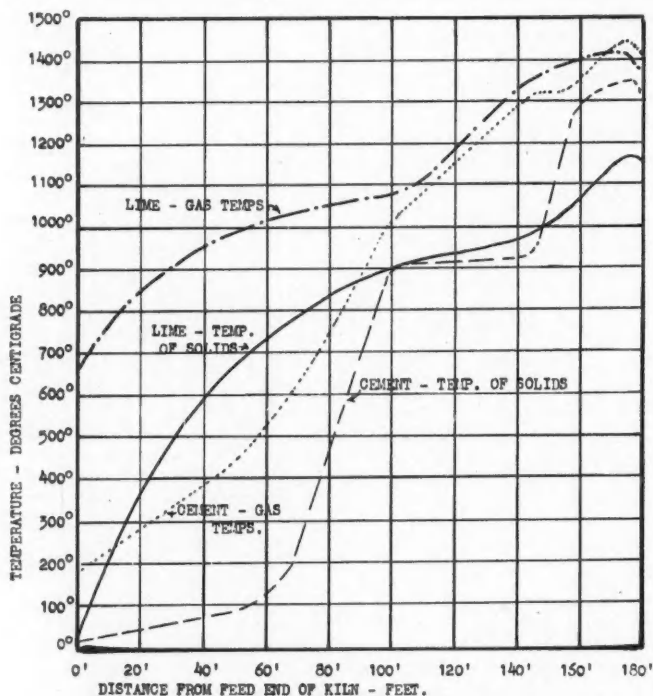


Fig. 5.—Rotary Kiln Temperatures—Lime and Cement.

cement and aluminous cement, but their very limited use for this purpose, to which they are much better adapted than to lime burning, is not an encouraging portent of their use for the latter purpose.

The Rotary Kiln.

Rotary kilns have been used for lime burning for nearly as long as for making Portland cement, but it is only in the last twenty years that their commercial success in the United States has been sufficiently marked to warrant their widespread use in lime production. The cost of making lime in rotary kilns—even large ones—is higher than in efficient shaft kilns, and when small rotary units are considered the position is still less favourable. Few lime plants in England are at present large enough to install a rotary kiln of the size common in the United States, that is, with a capacity of upwards of a hundred tons a day.

Rotary kilns used for lime are practically the same in general design as those used for Portland cement, but the process to be carried out is rather widely different. In burning cement by the wet process the heat is used up fairly evenly throughout the kiln and there is an exothermic reaction—the combination of lime with silica and alumina—near the discharge end which speeds up the process there and beneficially influences the efficiency of the kiln. Furthermore, the calcium carbonate which has to be decomposed is in very finely divided form and is therefore calcined with extreme rapidity once it reaches decomposition temperature.

In burning lime the usual practice is to feed stone of pebble size, and therefore heat exchange is slower and there is normally a much greater difference of temperature between gases and solids in the kiln. In the burning zone, per unit of output, there is much more calcination to perform and the calcination time is very much longer than that of the fine dust in a cement kiln. Moreover, there is no exothermic effect in the lime kiln to boost the heat at the hot end. The difference between the two processes is illustrated in *Fig. 5*, which shows the approximate temperature conditions (temperatures of solids and gases) in a rotary kiln 180 ft. long when burning lime and when burning cement. The output, when burning cement, is much greater than with lime.

Rotary kilns burning pure limestone ordinarily have efficiencies of only between 25 and 35 per cent. (about 2.3 to 3.3 tons of lime per ton of coal of 13,000 B.Th.U.s). The low efficiency, in spite of the use of small stone, is due mainly to the very poor conditions for the transference of heat from gases to solids. The surface exposed to the hot gases is only a very small fraction of the exposed surface in a shaft kiln of the same output burning lumps, and the time of passage through the rotary kiln is of the order of only one-twentieth of the time in a shaft kiln. There is relatively little turbulence in the gases passing through a rotary kiln, and consequently the upper layers pass their heat to the stone mainly by radiation. This stratification effect has been proved by Azbe in the United States by analysis of the gases at different levels of a rotary kiln near its discharge end, the carbon dioxide in the gases at the highest level being 14 per cent. and at the lowest level, just above the stone, 32 per cent., whilst at intermediate positions the carbon dioxide varied progressively between these limits.

The time that the lime is in the burning zone of a rotary kiln is so short that any wide variation in size of the stone fed is most undesirable. The size often used is $\frac{1}{2}$ in. to $1\frac{1}{2}$ in., and one of the difficulties met with in actual operation is the dissociation of the last core of stone in the larger pieces, which occupies an altogether disproportionate length of the kiln and thereby reduces the efficiency and capacity of the plant. There have been several attempts to meet this difficulty. One is to have a lip at the discharge end of the kiln so as to keep back a heavy charge at this end and thus give the lime a longer time of passage without increasing the length of the kiln. There are difficulties in retaining a quantity sufficient to be of value, and the device has not always proved effective in practice. Another suggestion is to discharge the lime into closed insulated

refractory-lined bins where the excess heat in the fully-burnt lime will serve to calcine the cores left in the partially-burnt lime. For example, if the fully-burnt lime is heated before discharge to an average temperature of 1100 deg. C. it will contain $200 \times 0.21 = 42$ C.H.U.s per pound of lime of heat available for calcining unburnt core, and this amount is sufficient to calcine $\frac{42}{38.5} = 1.1$ lb. core, or about 10 per cent. of the total output. The idea is therefore theoretically sound, but in practice it involves costly and cumbersome equipment which is rendered still more unwieldy if, after "soaking," the hot lime is to be cooled by the air for combustion so as to recover that heat in the usual way. A better solution of the difficulty is to feed closely-graded stone so that it burns evenly.

It might be thought that so long as the stone was less than a certain size—small enough to be calcined in the time available—uniformity would not matter and that all stone below that size could be fed to the kiln together. In practice it is found that such a mixture gives very poor results. The less open burden reduces heat exchange from gases to solids, and the fine stone tends to remain near the centre of the mass that is turning over in the kiln and is not exposed to the heat, so that actually fine stone may pass through unburnt.

A plain horizontal cylinder is a poor preheater of stone and lime cooling unit. Cement clinker coolers are fitted with various devices for lifting and cascading the clinker to improve heat exchange, and the same kind of cooler may be used for lime. For the same reason the preheating zone of a wet-process cement kiln is often fitted with chains to expose a large surface of the slurry to the hot gases. The same device is not applicable to a lime kiln, but the feed end could be designed with lifting plates or any of the numerous devices employed in rotary driers to improve heat exchange. In this way the length of the kiln could be reduced by a decrease in length of the preheating zone, and the cost correspondingly reduced and the overall efficiency increased. However, when everything possible is done in this way the rotary kiln remains a relatively inefficient lime calciner, and it is only by improving the heat exchange in the actual burning zone that its efficiency may be raised to any great extent. At the high temperature necessary, any complications in the design of the lining introduced with a view to lifting and cascading the lime are liable to be more trouble than they are worth. Something might be done to increase the turbulence of the flow and avoid stratification, but, beyond this, close grading and a higher flame temperature would seem to offer the best chance of improved efficiency. If the lime is of uniform size it is possible to operate at very high temperatures without causing over-burning, and the temperature is, in practice, then limited only by the properties of the refractory lining. Refractories for kilns in general are to be considered in a subsequent article.

Some rotary lime kilns are fired with powdered coal in the same way as cement kilns, but the ash tends to form a sintered coating on the surface of the lime pebbles which not only decreases their purity but makes their slaking unsatisfactory unless they are crushed first. In the United States gas firing, with producer or natural gas, is therefore used when lime of the highest quality is desired.

Shaft Kilns for Small Stone—Gas Fired.

We have seen that, so far as the calcination of the stone is concerned, a short kiln operating on small stone will do the work of a tall one burning large stone. We have now to consider how far this advantage of small stone can be made use of in a commercial plant embodying shaft kilns. Two kinds have to be considered, gas-fired and mixed-feed, and they will be discussed in that order.

The general design of the shaft of a gas-fired kiln is controlled mainly by three factors, (1) the time required for calcination, preheating, and cooling, which affects the volume of these zones and of the whole kiln, (2) the space required for the combustion of the gas, which affects the size of the burning zone, and (3) the distance the gas will travel horizontally through the voids before and during combustion, which controls the position of the gas ports and the size and shape of the cross-section of the kiln at this point. These factors will be more fully dealt with when the design of gas-fired kilns is discussed in a subsequent article. They concern us here only in indicating whether, in fact, a gas-fired kiln is practicable for burning small stone.

The distance that gas and flame will penetrate horizontally through the voids in a vertical shaft kiln charged with stone of even size is proportional to the size of the stone. This is a natural corollary of the fact that the resistance is inversely proportional to the size of the stone. Experience has shown that the maximum diameter for reasonably even burning in a gas-fired kiln charged with 6-in. stone of even grading, when the gas is admitted at ports flush with the side walls, is about 9 ft., so that the distance of penetration of the gas is 4 ft. 6 in. With the mixed stone ordinarily charged to a kiln the maximum diameter is less, but on the other hand, as will be seen subsequently, there are methods of operation which enable greater distances to be penetrated. On the basis of a penetration of 4 ft. 6 in. for 6-in. stone the maximum distance of flame penetration for 2-in. stone is 18 in., and for 1-in. stone it is 9 in. It is obvious, therefore, that if gas-fired kilns are to be used for small stone the cylindrical form at the burning zone is impracticable.

The cylindrical form of kiln is the cheapest to build, the best form mechanically, and the form which involves least thermal loss through the walls, and consequently it is preferable to maintain that form in the preheating and cooling zones of the kiln. To reduce the distance through which the flame has to travel the section of the kiln may be changed to long and narrow at the gas ports as in *Fig. 6*, which shows the shape of such a kiln at the gas inlet section when designed for 2-in. stone and of capacity between one and one-and-a-half tons of lime an hour. This shape of gas-fired kiln, but with less constriction, is already used for burning larger stone. The obvious objection is that the constriction will tend to pack the lime together and to make it "stick" or "arch" a few feet above the gas ports. To avoid this disability the oblong section may be continued upwards to a point above the burning zone, where sticking is no longer a danger, but the lengthening of the narrow section entails much greater resistance to draught (and consequently greater power consumption) and a

greater height of kiln, because the burning zone is lengthened. The tendency of lime to stick in a kiln depends largely on the maximum temperature in the burning zone and, as already mentioned, the temperature is less when burning small stone than in a kiln burning large stone. With proper control, therefore, sticking may not be a difficulty in a kiln of the form shown in *Fig. 6* and operating on 2-in. to 3-in. stone, while the lime will be more uniformly and lightly burnt.

The space required for the actual combustion of the gas remains to be considered. In any gas-fired kiln a gas giving a long flame is preferred, so as to have as long a zone as possible where the temperature exceeds calcination temperature without at any point becoming too high. With small lime the greater surface exposed and the smaller voids increase the rate of combustion and thereby reduce the space required for combustion, but the reduction is not

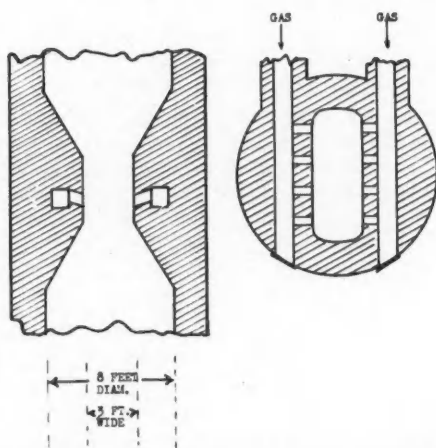


Fig. 6.—Gas Inlet Zone of Kiln for Burning 2-in. Stone.

by any means proportional to the reduction in the size of stone. The burning zone of a gas-fired kiln burning small stone cannot, therefore, be reduced in proportion to the space required for calcination itself.

From a full consideration of all the various factors, which would require too much space to be set out here, the conclusion is reached that a gas-fired shaft kiln is practicable for stone of 2 in. and over, but that below 2 in. the distance the gas spreads is so short that the necessary constriction and complication of the gas inlet zone become excessive. The necessary height of a kiln for 2-in. stone is about one-half of the height of a kiln for 6-in. stone, the greater length than calcination considerations alone would indicate being due to constriction at the gas ports and the combustion space required for the gas. The stone will be more uniformly and lightly burnt than large stone, probably with a somewhat lower fuel consumption. The power required for exhausting the kiln will be

greater than in a kiln of equal capacity using large stone. These tentative conclusions still lack confirmation by a practical trial on a large scale.

For stone smaller than 2 in. the shaft kiln is unlikely ever to be suited to gas-firing, except possibly in the modified form similar to the louvre dryer. With modern heat-resisting metals louvre dryers may now be used up to temperatures not far removed from those of lime burning, but higher temperatures would require still further progress in heat resistant metals or in mechanically strong refractories.

Shaft Kilns for Small Stone—Mixed Feed.

In a mixed-feed shaft kiln there is nothing to limit the size of stone that may be burnt. The heat is produced by the combustion of the fuel amongst the lime, and if the length of the burning zone is to be limited to agree with the requirements of the rate of calcination it is necessary that the fuel should be burnt at such a rate as to be completely consumed in the burning zone. This involves the use of fuel of a size less than the usual by an amount corresponding to that of the stone, and a similar size reduction is demanded by heat exchange considerations. In a mixed-feed kiln in which the fuel is mixed evenly with the stone, and not placed in layers, the best results are attained when each piece of fuel is surrounded by pieces of lime and vice versa, which involves there being at least as many pieces of fuel as pieces of lime. If the fuel-to-lime ratio is assumed to be 1 to 5, each piece of fuel should therefore weigh not more than one-fifth of the average weight of the lime. The average fuel size should therefore be not more than the cube root of 0.2 times the limestone size, that is to say, not more than 0.6 of the stone size. If the limestone is 1 in. the fuel should therefore be 0.6 in. or less, but to be burnt completely in the short burning zone most types of fuel would have to be considerably smaller than this, and ordinarily there is a price advantage in using small fuel.

The rate-of-calcination curves in *Fig. 4* show that 1-in. stone requires one-eighth the time of 6-in. stone. The effective height of an efficient mixed-feed kiln for 6-in. stone may be taken as 60 ft., so that 1-in. stone will require about 7.5 ft. A kiln to produce a ton of lime an hour would therefore have a shaft 7 or 8 ft. in diameter and 7 ft. 6 in. high, not including the height occupied by the charging and discharging arrangements. If still smaller stone, say $\frac{1}{2}$ in., is to be burnt the height will be much less. It will be realised that there are considerable difficulties in arranging feeding and discharging equipment for a kiln that is so short in relation to its diameter, and in ensuring evenness of draught.

The dimensions of a kiln for stone 1 in. or less approach those of a gas producer, and the air distribution, charging, and discharging arrangements of a cylindrical gas producer are some guide to the requirements of a kiln. In the kiln, however, the difficulties are greater because the limestone and fuel have first to be evenly mixed and then fed to the kiln without segregation, which is more difficult than feeding fuel alone to a producer. Moreover in discharging the lime it is much more important to draw it evenly over the whole area than when discharging the ash from a producer, and the volume of lime is enormously greater than

the volume of ashes. In one form of kiln that has been proposed the stone and fuel are spread over the level surface at the top of the kiln by means of a revolving feeder, and there is a continuous discharge from the bottom by a special form of revolving grate. The discharged lime falls into a closed hopper bottom whence it is drawn off as required, and the draught is provided by blowing air into this closed bottom and thence through the grate and the kiln shaft.

On account of the favourable heat exchange conditions it is possible to attain high fuel efficiency without hard-burning the lime and without the danger of sintering the fuel ash on to the surface of the lime. Furthermore, in so short a shaft the lime is not crushed and there is ordinarily no tendency for it to fracture during heating up. Even chalk, which is notoriously easily fractured by too rapid heating, when in 1-in. pieces may be heated rapidly without breaking down to smaller size. As a consequence it is very easy to separate the ashes from the lime merely by grading the product from the kiln on a screen with openings smaller than the smallest stone and larger than the largest fuel size. It is obvious that hand sorting is impracticable on such small lime, and therefore to produce a high-grade lime it is necessary that the stone fed should be sufficiently pure, that the lime be thoroughly burnt, and that fuel ash be efficiently separated. Provided the difficulties of design of charging and discharging plant can be surmounted there is no doubt that pure lime can be produced in this way at low capital and running costs, and even a partial success would provide cheap lime suitable for hydration. The calcination of small stone is, in fact, the field to which the mixed-feed kiln is best suited, and it is the logical outcome of the natural properties of limestone.

It may well be suggested that the case for the calcination of small stone is built up from data obtained in small-scale tests, which might be considered inconclusive. A solution of the mechanical difficulties of maintaining an even bed and even draught on a short kiln has not yet been tried on a large scale, but the actual calcination of small stone in short kilns at low temperatures has been carried out with confirmatory results. Moreover the process, without mechanical elaboration (and consequently with high labour requirements), is in operation in South America burning sea shells to lime.

(To be continued.)

BINDING CASES FOR "CEMENT AND CEMENT MANUFACTURE"

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Research on Cement and Lime.

THE following notes are taken from the Report of the Building Research Station for the year 1935, just published by H.M. Stationery Office (price 3s. 6d. net).

Specification for Mortars.

A committee which considered a proposal to prepare a standard specification for mortars came to the conclusion that the proposal was impracticable. The difficulty is partly technical and partly economic. Lime has been considered by many committees of the British Standards Institution, and after nine years the specifications are not yet issued. The properties of a lime depend upon its source and it is almost impossible to draw up a national specification which will rigidly define the useful and the useless for a purpose such as mortar. Ideal conditions may be imagined and conditions laid down for their fulfilment, but when this is done it often happens that the specification has the effect of excluding certain sources of limestone despite the fact that the limes made from such stones have for generations been used satisfactorily for mortars. There remains the possibility of developing a national standard in terms of tests of the performance of a mortar rather than of its composition. This possibility is still being pursued and it is hoped that some solution will be found on these lines.

The Constituent Compounds of Set Cements.

Calcium Sulpho-aluminates.—Examination is being made of the solubility equilibria of the high sulphate form of calcium sulpho-aluminate in solutions of sodium chloride and sodium sulphate at 25 deg. C. and in water at temperatures above 25 deg. C. The equilibrium in salt solutions has a direct bearing on some problems arising from the attack of sea-water and sulphate salts on concretes, while the study of the solubility in water at higher temperatures should provide data for a fuller understanding of accelerated tests for the soundness of cements. While some progress has been made, it has not been possible to complete this investigation owing to pressure of other work. It has previously been reported that an octahydrate of the high sulphate form of calcium sulpho-aluminate is formed when this salt is maintained in an atmosphere of sufficiently low vapour pressure. The density of this octahydrate, determined in ethyl alcohol, has been found to be 2.43 at 25 deg. C. This is considerably higher than the value of 1.73 found for the normal hydrate.

Hydrated Calcium Silicates.—The examination of the properties and the phase relations of the hydrated calcium silicates has been continued. It has been found that, at ordinary temperatures, lime solutions in the range of concentration from 0.006 to 0.11 gm. CaO per 100 cc. are in equilibrium with a series of gelatinous solids varying continuously in composition from $\text{CaSiO}_3 \cdot x \text{H}_2\text{O}$ to approximately $3 \text{CaO} \cdot 2\text{SiO}_2 \cdot y\text{H}_2\text{O}$. It seems that this must be regarded as a solid solution series. The variation in composition of the solid phase with the liquid composition is shown in Fig. 1. The curve given is the mean of four curves obtained with different materials, two prepared from sodium silicate solutions

by precipitation with calcium nitrate and lime-water and two by the slow interaction of finely-ground silica gel and calcium hydroxide.

The composition of the calcium silicate gel existing at concentrations between 0.11 per cent. CaO and that of saturated lime-water (approximately 0.12 per cent. CaO) has not been determined. The very narrow range of concentration and variations in the apparent solubility of lime with the fineness of the crystals make the examination difficult. Similar curves have been obtained for the relation between the lime concentration of the solution and the composition of the solids using tricalcium and dicalcium silicates as the starting materials, but the lime content of the solid is always high owing to the difficulty of attaining complete hydration of the initial materials.

The crystalline compounds prepared from $3 \text{ CaO} \cdot \text{SiO}_2$ and $2 \text{ CaO} \cdot \text{SiO}_2$ by hydration in high-pressure steam have been further examined. The hydrated

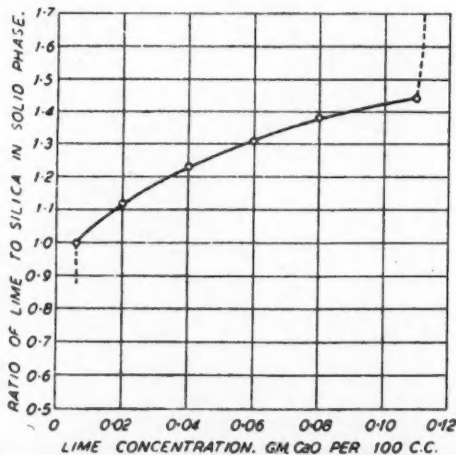


Fig. 1.—The relation between the Lime : Silica Ratio of the Solid and the Solution Concentration in Hydrated Calcium Silicate gels in contact with Lime Solutions.

tricalcium silicate mentioned in the previous report has a composition expressed by the formula $3 \text{ CaO} \cdot \text{SiO}_2 \cdot 2 \text{ H}_2\text{O}$ when dried over saturated lead nitrate solution. It occurs as very minute needles, whose form and symmetry have not yet been determined, with a mean refractive index of 1.587 and a birefringence of about 0.003. The relation of this compound and the crystalline dicalcium silicate hydrates, of which there appears to be more than one form, to the amorphous hydrated silicates discussed is not yet clear, and the study of these is being continued.

Hydration of Cement.

Some attention has been paid to the possibility of following the hydration process in cement by the preparation and microscopic examination of thin

sections of the set cements. Sections have been cut from 1-in. cubes made from different cements and stored for varying periods. Some difficulty was at first experienced in preparing sections but, by modifications in the grinding method and by grinding the section to a thickness of 10-15 μ , suitable specimens were obtained for examination under the microscope. Some attempts were also made to stain the sections selectively with organic dyes, and Naphthol Green B has been found useful in this respect, a green stain being obtained on the gel with the unhydrated material left unstained. The technique and results have been published.¹

Fineness of Portland Cement.

The problem of determining the fineness of Portland cement powder has become more necessary because the fineness to which cements are now ground has rendered the ordinary test for residue on the British standard 100 and 170 sieves of little value as a measure of fineness, and because of the efforts now being made to relate the chemical compound composition of cement with the properties to be expected from it. Since the initial stages of the hydration of Portland cement depend on a surface reaction between the water and the cement, the most rational measurement of fineness should be a determination of the amount of surface per gram of cement, or "specific surface." An apparatus has been constructed for this purpose which is practically identical with one described by Wagner.² The method depends on a sedimentation of the powder dispersed in kerosene, a measure being made of the turbidity of the suspension after definite times by means of a photo-electric cell. The method is comparatively rapid and gives reproducible results when a standard practice is adopted. Preliminary tests have been carried out chiefly on the method of dispersing the cements and on the relation between the surface area determined in this way and that calculated from microscopic measurements.

Special Cements.

The rise in temperature in mass concrete, produced by the heat evolved during hydration of the cement, is considerable and leads to the formation of shrinkage cracks when cooling subsequently occurs. Dams are also often exposed to the action of very pure or slightly acid waters draining from mountain and moorland areas. Such waters tend to dissolve the lime from a cement and progressively increase the permeability of the concrete mass. Action of this type does not occur on well-made concrete of very low permeability, but with concretes of lower quality water may permeate slowly into the mass and solution of the cement may occur. The use, if practicable, of a cement more resistant than the normal to this solvent action would afford a valuable safeguard in practice.

Heat Evolved during the Hydration of Cement.—Laboratory methods of a somewhat elaborate type are available for measuring and comparing the heat

¹ Parker, T. W., and P. Hirst. The preparation and examination of thin sections of set cements. *Cement*, 1935, 8 (10), 253-41. (*B.S.A.*, 1935, No. 1608.)

² Wagner, L. A. A rapid method for the determination of the specific surface of Portland cement. *A.S.T.M. Proc.*, 1933, 33 (2), 553-70. (*B.S.A.*, 1934, No. 538.)

evolution of cements, but the production of a simple test apparatus which could be more generally used was very desirable. As a result of the investigations made for the British Sub-Committee on Special Cements, a simple adiabatic calorimetric apparatus has been produced. Comparative tests have been made in this calorimeter and the standard type of calorimeter described in the Report for the year 1931 which has been in use at the Building Research Station for some years, and have shown that the simplified form gives sufficiently accurate results for practical purposes. A comparison of the heat of hydration of cements has been made using this apparatus, the standard Building Research Station adiabatic calorimeter, and the heat of solution method used in the U.S.A. The test in the first two cases is carried out on a 1 : 6 (weight) concrete mix and in the third case on the neat cement. Some results obtained are shown in Table 1.

TABLE 1.—COMPARISON OF MEASUREMENT OF HEAT OF HYDRATION OF CEMENTS BY DIFFERENT METHODS.

Cement no.	Heat evolution (calories per gram cement).							
	Standard adiabatic calorimeter.			Simplified adiabatic calorimeter.			Heat of solution method.	
	1 day	3 days	7 days	1 day	3 days	7 days	7 days	28 days
207	47.4	76.0	88.1	50.0	76.6	86.4	95.9	99.7
208	31.9	57.0	67.4	34.8	53.8	66.5	72.0	113.9
210	38.6	62.3	74.3	43.7	63.2	74.3	79.4	94.4
220	21.2	32.9	37.7	20.1	34.4	42.4	65.2	79.7
209	22.8	50.2	63.2	24.1	54.0	66.5	68.9	102.4
212	25.9	48.7	58.9	26.6	49.6	62.3	62.1	87.5

The results given in the table show that the simplified adiabatic calorimeter gives results in close agreement with those obtained from the standard adiabatic calorimeter and that it forms a satisfactory apparatus for test purposes. This simplified adiabatic calorimeter has been provisionally adopted by the International Sub-Commission on Special Cements as an agreed test method. The heat of solution method gives somewhat higher results than the adiabatic methods with cements of normal heat evolution, and considerably higher results with cements of low heat evolution (see No. 220 in Table 1). This difference is attributable to the difference in the average temperatures at which the test samples are cured in the adiabatic and heat of solution test methods.

Thermal Constants of Setting Concrete.—The evolution of heat by concrete during the process of setting has been under investigation for some years past by the method of adiabatic calorimetry,³ while the temperature variations to be expected in certain typical problems involving the placing of large masses of concrete have been examined both practically and mathematically.⁴ Throughout this work it has been necessary to assume values for the thermal constants of the

³ Davey, N. Temperature rise of concrete. *Concrete Constr. Eng.*, 1931, 26 (10), 572-5. (*B.S.A.*, 1931, No. 2055.)

⁴ Davey, N., and F. N. Fox. Temperature rise in hydrating concrete. *Building Research Technical Paper No. 15*. London, 1933. H.M. Stationery Office.

concrete, notably for the specific heat and the thermal diffusivity; and, in the absence of other data, figures for specimens three days old have had to be used. Since there is likely to be some initial variation in these quantities, due to the fixation of free water and the accompanying chemical changes, methods have now been devised which permit measurements to be made during the actual period of setting. The thermal conductivity and thermal diffusivity have been found experimentally and the specific heat has then been computed from these quantities and the density of the material. The results for a typical specimen (1 cement : 6 sand : 0.6 water by weight) made with normal Portland cement are shown in Table 2. It will be seen that both the thermal conductivity and thermal

TABLE 2.—THERMAL CONSTANTS OF NORMAL PORTLAND CEMENT MORTAR HAVING A DENSITY OF 2.11

Time after mixing (hours)	Temperature (deg. C.)	Conductivity k (C.G.S.)	Diffusivity h^2 (C.G.S.)	Specific heat $s = k/h^2C$
4	18.6	0.00578	0.0110 ₄	0.248
6½	19.8	0.00578	0.0110 ₇	0.247
25	33.0	0.00573	0.0110 ₉	0.245
31	35.1	0.00575	0.0110 ₉	0.246
48½	40.0	0.00581	0.0112 ₅	0.245
55	41.1	0.00582	0.0112 ₄	0.245
72	43.3	0.00586	0.0113 ₅	0.244

diffusivity increase slightly with time, while the specific heat shows a slight decrease. Fortunately the changes in all these quantities are small enough to be ignored in any approximate mathematical analysis. This is generally true for all the mixes studied. It may be noted that where great accuracy is not required the specific heat of freshly mixed concrete can be computed from the specific heats of the ingredients. In the example, assuming values of 0.19 for the sand and 0.20 for the cement, a value between 0.25 and 0.26 is obtained for the concrete, which is in good agreement with the experimental figures.

Pozzolanas.

Two accelerated tests which it is thought may provide a measure of the efficiency of the pozzolanas present in a pozzolanic cement have been examined. In the first of these tests, twelve briquettes of a 1 : 3 plastic mortar are cured for one day in moist air at 18 deg. C. and then split into two groups of six. One set is cured further for two days in water at 18 deg. C. before testing, and the other set cured for 24 hours in water at 50 deg. C. followed by one day in water at 18 deg. C. The ratio, or difference, between the two strength values obtained is found to be dependent on the amount and quality of the pozzolana present. The second test is essentially a measure of the ability of the pozzolana present in a pozzolanic cement to combine with the free calcium hydroxide liberated in the setting of the Portland cement fraction of the cement. The pozzolanic cement is gauged neat with water to a defined plastic consistency, cured for three days in moist air at 18 deg. C. followed by four days in water at 50 deg. C. The

specimen is crushed, dried *in vacuo* over sulphuric acid, and ground to pass a British standard 100-mesh sieve. The amount of lime which can be extracted by shaking 1 g. with 200 c.cm. lime-water (0.06 g. CaO per 100 c.cm.) for 24 hours at laboratory temperatures is then determined. The method does not afford a strict measure of the amount of free calcium hydroxide present in the set cement.

The use of curing temperatures of 50 deg. C. in both tests is open to some objection since the activity of pozzolanas increases rapidly with temperature, and not necessarily at the same rate for different materials. A long series of tests has, however, shown that the two methods do, in practice, afford a means of differentiating between cements containing efficient and inefficient pozzolanas. The term "efficient" is used to denote the combined effect of the quality and amount of the pozzolana present in a pozzolanic cement. These tests must be regarded as supplementary to the normal strength tests carried out to determine whether the strength of the cement at early ages conforms to the required standards.

Data are shown in Table 3 on pozzolanic cements composed of 60 per cent. of one Portland cement and 40 per cent. of various pozzolanas. In addition to the results of the two tests already described, the relative strengths developed in plastic mortars at an age of six months and the resistance of the mortars to

TABLE 3.—TESTS ON POZZOLANIC CEMENTS (40 PER CENT. POZZOLANA, 60 PER CENT. PORTLAND CEMENT)*

Pozzolana.	Lime extraction test (per cent. CaO ex- tracted).	1 : 3 Mortar.				1 : 3 plastic mortar. Tensile strength at 6 months expressed as percentage of that of the 1 : 3 mortar with Port- land cement alone at the same age.	Resist- ance to attack by sulphate salts.	
		Tensile strength (lb. per sq. in.)		Ratio. B/A	Differ- ence. B—A			
		18° (A)	50° (B)					
Burnt clay	A	4.6	144	213	1.48	69	81	Low Low Low
	B	4.6	199	248	1.25	49	84	
	C	5.5	128	155	1.21	27	80	
	D	3.8	149	207	1.39	58	91	Inter- mediate
	E	3.2	193	382	1.98	189	91	
Spent	F	4.0	158	305	1.93	147	89	High
	shale G	2.7	158	319	2.02	161	93	High
Rhenish	trass H	4.2	156	324	2.08	168	86	High
None		8.3	246	308	1.25	62	100	Low

* The same Portland cement was used as the base cement throughout this series.

attack by sulphate salt solutions (sodium and magnesium sulphates) are shown. The series of pozzolanas given includes materials of good and inferior quality as judged from the results of the sulphate resistance tests and the six months' strength tests. The ratio of the strength values obtained by curing at 50 deg.

and 18 deg. C. differentiates clearly between the good and inferior materials in this series, but the division given by the lime extraction test is less definite. When different Portland cements are used as the base cement for blending with the pozzolanas, the division obtained by this 50 deg. C./18 deg. C. strength ratio is less clear cut, but nevertheless it seems likely to afford a method of test which is fairly satisfactory and superior to any other known short-period test. The lime extraction test will probably provide a useful additional control test.

The testing of pozzolanas, as distinct from pozzolanic cements, involves blending them with either Portland cement or hydrated lime. In the former case the tests discussed here can be used, while for the latter case early-age strength tests on plastic 1 : 2 : 9 hydrated lime : pozzolana : standard sand mortars cured both at normal temperatures and 100 deg. C. seem likely to be adequate as a measure of the value of the pozzolana in a lime-pozzolana mix. The extent, however, to which tests on lime-pozzolana mixes can be applied, as is done, for instance, in certain Continental specifications, to assess the value of a pozzolana for use in Portland cement mixes is not yet entirely clear. It may prove preferable for this purpose to rely on tests on mixtures with a Portland cement.

Tests are being carried out in co-operation with cement manufacturers to ascertain the degree of reproducibility of the various tests when carried out in different laboratories. A series of tests has also been made during the year on the heat of hydration of pozzolanic cements using the adiabatic calorimetric method.

Renderings.

The troubles associated with external renderings may be broadly grouped under two main headings, (a) structural failure (i.e., detachment of rendering from the backing), and (b) crazing (i.e., deterioration of appearance by development of hair cracks). Failures under the first heading seem to be generally associated with the presence of soluble salts in the backing and the maintenance of damp conditions in the wall. The detachment of the rendering may result from the direct crystallisation of the salts or from the crystallisation of compounds resulting from an interaction between these salts and the rendering. An attempt is being made to develop a rendering which will allow these salts to pass freely to the surface.

As a result of the examination of full-scale exposure panels there is a definite suggestion that crazing can be considerably reduced by using a weaker mix than the normal cement-sand mix. This can be accomplished conveniently by dilution of the cement with lime. The effects of the method of applying the rendering and the process of finishing are also being investigated. In this connection a study is being made of Continental technique.

With regard to the protection afforded against rainwater by an external rendering, preliminary experiments have shown clearly that a porous rendering may afford excellent protection from weather, but that an impervious rendering that has cracked is definitely inefficient.

Limes and Plasters.

Specifications for Building Limes.—The outstanding problem in the specification for building limes is that of the method of testing hydraulic strength. While test results can be reproduced at the Station with satisfactory accuracy, difficulties have been experienced in obtaining good agreement between the results of tests in different laboratories. Most of the tests have been made with dry hydrates, and it was found that some of the discrepancies were likely to be due to slight "unsoundness" in the lime. The degree of "unsoundness" here considered is too slight to be of importance in practice, but the necessarily artificial method of rapidly developing the strength causes it to have a noticeable effect. The test method was therefore altered to include two days' damp storage in the moulds before steaming. A combined test was then carried out in five laboratories simultaneously, six commercial samples of hydrate being tested in each. The results showed that while the different testers were able to obtain consistent results yet in certain cases there was a systematic error, due presumably to a variation in mixing which affected the water-ratio. The problem is being further investigated.

Magnesian Limes.

The high magnesian limes present especial difficulties in testing, particularly in testing "soundness," since as usually slaked (or rather dry hydrated, in a large pile covered with sand) on the building site the product still contains a variable amount of "hard-burnt" very slow-slaking material. Whether this will ultimately slake and expand in the wall depends on the degree of "hard-burning" and on the temperature and humidity conditions prevalent in the wall over long periods. A rapid intensive steam curing of such limes will almost always result in a marked expansion, due to further slaking of a portion of this "hard-burnt" material. Evidently less severe curing conditions must be used in testing high magnesian hydrated limes.

Strength of Brickwork built in Lime Mortar Gauged with Cement.

The tests to determine the strength of brickwork built with lime mortars gauged with cement have been extended to study first a change in the type of lime used and, secondly, a change in the type of sand. In the series of tests previously reported dry hydrated lime and a pit sand with rounded grains were used. In the more recent tests the effect of using lime putty with the cement and sharper river sand of similar grading have been examined.

The composition of the mortars varied from 1 : 2 to 1 : 4 by volume, the ratio of lime to cement being 0 : 100, 70 : 30, 75 : 25 and 80 : 20. The piers, which measured 9 in. by 9 in. by 36 in., were stored at 64 deg. F. for three months, when crushing and deformation under load were measured. In Table 4 are given the results of tests on piers built with lime : cement : sand mortars, using lime putty, normal Portland cement and pit sand through $\frac{1}{4}$ -in. mesh. In Table 5 are given the results from piers built with mortars composed of dry hydrated lime, normal Portland cement and sharp river sand of similar grading. The tests are not sufficiently advanced to draw detailed conclusions, but the results

TABLE 4.—CRUSHING STRENGTH OF BRICK PIERS BUILT IN LIME MORTAR GAUGED WITH PORTLAND CEMENT.

Materials: Lime putty, pit sand and Portland cement. Dimensions of piers 9 in. by 9 in. by 36 in.—four courses to 1 ft. All tests made when mortar was 3 months old. Crushing strength of bricks 2,545 lb. per sq. in.

Series C.	Ratios by volume.		Water- cement- ratio by weight.	Crushing load on pier (tons).			Value of E at a stress of 500 lb. per sq. in. (lb. per sq. in.).	Mean crushing strength of pier.		Mean strength of mortar (lb. per sq. in.).	
	Cement : lime.	Cement + lime : sand.		I.	II.	Mean of I & II		(Tons per sq. ft.).	(lb. per sq. in.).	Compression.	Tension.
1 C	100 : 0	1 : 3	0.75	39.6	42.3	41.0	0.49×10^6	72.8	1130	1630	239
2 C	30 : 70	1 : 3	1.40	37.5	40.2	38.9	0.42×10^6	69.2	1070	164	39
3 C	25 : 75	1 : 3	1.53	33.0	34.7	33.9	0.43×10^6	60.2	935	78	22
4 C	20 : 80	1 : 3	1.71	27.2	33.3	30.3	0.29×10^6	53.8	835	60	14
5 C	25 : 75	1 : 2	1.21	34.0	32.5	33.3	0.35×10^6	59.2	918	180	36
6 C	25 : 75	1 : 4	2.01	32.7	35.4	34.1	0.30×10^6	60.6	940	47	12

TABLE 5.—CRUSHING STRENGTH OF BRICK PIERS BUILT IN LIME MORTAR GAUGED WITH PORTLAND CEMENT.

Materials: Dry hydrated lime, river sand, and Portland cement. Dimensions of piers 9 in. by 9 in. by 36 in.—four courses to 1 ft. All tests made when mortar was 3 months old. Crushing strength of bricks 2,545 lb. per sq. in.

All tests made when mortar was 3 months old. Crushing strength of bricks 2,545 lb. per sq. in.

Series D.	Ratios by volume.		Water- cement- ratio by weight.	Crushing load on pier (tons).			Mean crushing strength of pier.		Mean strength of mortar (lb. per sq. in.).	
	Cement lime.	Cement + lime : sand.		I.	II.	Mean of I & II	(Tons per sq. ft.)	(lb. per sq. in.)	Compression. Tension.	
									Compression.	Tension.
1 D	100 : 0	1 : 3	0.80	51.0	52.3	51.7	92.0	1430	2760	421
2 D	30 : 70	1 : 3	1.44	39.5	38.3	38.9	69.0	1080	386	95
3 D	25 : 75	1 : 3	1.48	37.8	42.0	39.9	71.0	1100	287	77
4 D	20 : 80	1 : 3	1.58	37.6	38.9	38.3	68.1	1060	164	56
5 D	25 : 75	1 : 2	1.03	33.6	32.4	33.0	58.6	910	614	140
6 D	25 : 75	1 : 4	1.93	37.7	33.0	35.4	63.0	980	113	48

generally indicate that the reduction in strength due to a replacement of up to 50 per cent. of the cement by hydrated lime is negligible with bricks having a crushing strength similar to those used in this investigation.

Clay as an Aggregate.

A comprehensive investigation has been made of the suitability of a lightweight aggregate obtained by the heat treatment of clay. The aggregate bears a strong resemblance to expanded slate, a fact which is explained by the similarity in composition and method of treatment. It appears that the greatest obstacle to the commercial development of the slate aggregate is the cost of transport from the quarries, which are not readily accessible. If suitable deposits of clay are conveniently situated the prospects of this aggregate should be more favourable than those of slate. Chemical examination of the aggregate showed low soluble salt and sulphur contents. Partition slabs made with the aggregate conformed to the requirements of the British Standard Specification. A 1:3:8 *in situ* concrete gave good resistance to penetration of water under pressure and its crushing strength was quite adequate for column casing and panel filling. The transverse strength and abrasion resistance of paving slabs were within the limits of the British Standard Specification.

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